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L14: Entry 2 of 2 File: USPT Feb 26, 2002

DOCUMENT-IDENTIFIER: US 6350431 B1

TITLE: Compounds

Brief Summary Text (85):

Chemical functional groups in the surfactant molecules can be interconverted by chemical reactions well known to those skilled in the art. For example, a hydroxyl group can be converted to a methanesulfonic acid ester which can be treated with sodium azide and reduced to form an amine group. Carboxylic acid groups and ketones can be reduced to form alcohols, and alcohols can be oxidized to form ketones, aldehydes, and carboxylic acid groups.

Brief Summary Text (86):

Useful surfactant molecules are emulsifiers or detergents which can function as dispersing agents, wetting agents, <u>adsorbents</u>, anticaking agents, soil antiredispositioning agents, antistats, binders, carriers, pearlescents, conditioning agents, hydrotropes, defoamers, emollients, flocculants, humectants, lubricants, opacifiers, plasticizers, preservatives, release agents, scale inhibitors, stabilizers, suspending agents, thickeners, UV absorbers, water repellants, waxes, and polishes, and which contain at least one chemical functional group selected from the group consisting of an alcohol (OH), a nitrilo group including a primary amine (NH.sub.2) and a secondary amine (NH), a carboxylic acid (COOH), a sulfhydryl (SH), a phosphoric acid group, a phosphonic acid group, a phenolic group, a sulfonic acid group, a carbon-carbon double bond, and a ketone.

Brief Summary Text (167):

In addition, when a protein, peptide, peptoid or peptidomimetic can be chemically modified such as by partial oxidation to introduce an <u>aldehyde</u> group or a carboxylic acid group, a preferred "vector reactive group" can be selected from amino, aminoalkyl, aminoaryl, alkylamino, arylamino, hydrazino, alkylhydrazino, arylhydrazino, carbazido, semicarbazido, thiocarbazido, thiosemicarbazido, sulfhydryl, sulfhydrylalkyl, sulfhydrylaryl, hydroxy, carboxy, carboxyalkyl and carboxyaryl. The alkyl portions of the protein reactive group can contain from 1 to about 20 carbon atoms, and the aryl portions of the protein reactive group can contain from about 6 to about 24 carbon atoms.

Brief Summary Text (168):

An additional preferred vector reactive group can comprise a residue of a crosslinking agent. A useful crosslinking agent can react with a functional group such as, for example, an amine or sulfhydryl or carboxylic acid group or aldehyde group found in a linker and with a functional group such as, for example, an amine or sulfhydryl or carboxylic acid group or aldehyde group found in a vector or in a chemically modified protein or biological molecule such as described above. The residues of certain useful crosslinking agents, such as, for example, difunctional gelatin hardeners, bisepoxides and bisisocyanates become a part of, i.e., a linking group in, a vector-linker conjugate which is formed as a result of the crosslinking reaction of such a crosslinking vector reactive group with a chromophore. Vector reactive groups derived from various heterobifunctional cross-linking reagents such as those listed in the Pierce Chemical Company Catalog and handbook--Protein

Modification Section, (1994/5) are useful and non-limiting examples of such reagents include:

Brief Summary Text (180):

"Tissue" refers generally to specialized cells which may perform a particular function. It should be understood that the term "tissue", as used herein, may refer to an individual cell or a plurality or aggregate of cells, for example, membranes or organs. The term "tissue" also includes reference to an abnormal cell or a plurality of abnormal cells. Exemplary tissues include, for example, myocardial tissue (also referred to as heart or myocardium) including myocardial cells and cardiomyocites, membranous tissues, including endothelium and epithelium, laminae, connective tissue, including interstitial tissue, and tumors.

Brief Summary Text (182):

"Endothelial cells" or "endothelium" refers to an aggregate of cells and/or tissue which may be normal and/or diseased and which may comprise a single layer of flattened transparent endothelial cells that may be joined edge to edge or in an overlapping fashion to form a membrane. Endothelial cells are found on the free surfaces of the serous membranes, as part of the lining membrane of the heart, blood vessels, and lymphatics, on the surface of the brain and spinal cord, and in the anterior chamber of the eye.

Brief Summary Text (184):

"Epithelial cells" or "epithelium" refers to an aggregate of cells and/or tissue which may be normal and/or diseased and which may comprise one or more layers of cells that may be united together by an interstitial cementitious substance supported on a basement—membrane. Epithelium may be classified into various classes, including, for example, a single layer of cells (simple epithelium); more than a single layer of cells (stratified epithelium); and about three or four layers of cells that are fitted together substantially without the appearance of stratification. The different forms of simple epithelium are usually referred to as squamous, pavement, columnar, glandular, spheroidal and/or ciliated. Epithelium originates from the embryonic epiblast or hypoblast. Epithelium includes heart tissue, including infarcted heart tissue, cardiovasculature, the peripheral vasculature, such as arteries, veins, and capillaries, blood clots and the region surrounding atherosclerotic plaque.

Brief Summary Text (188):

Endothelial-leukocyte adhesion molecules (ELAM's) are antigens which are expressed by endothelial cells under conditions of stress which then facilitate the migration of the leukocytes across the endothelium lining the vasculature into the surrounding tissues. It is also the surprising discovery that these same endothelial leukocyte adhesion molecules may be advantageously exploited as receptors for targeting of chromophore polymers. These endothelial cell adhesion molecules belong to a family known as selectins in which the known members, such as GMP-140, all participate in endothelial leukocyte adhesion and include ELAM-1, LAM-1 and the granule membrane protein 140 (GMP-140) also known as platelet activationdependent granule-external membrane protein (PADGEM), VCAM-1/INCAM-110 (Vascular Adhesion Molecule/Inducible Adhesion Molecule) and ICAM-1 (Intercellular Adhesion Molecule). The cadherin family of cell adhesion molecules may also be used as targeting vectors, including for example, the E-, N-, and P-cadherins, cadherin-4, cadherin-5, cadherin-6, cadherin-7, cadherin-8, cadherin-9, cadherin-10, and cadherin-11; and most preferably cadherin C-5. Further, antibodies directed to cadherins, such as, for example, the monoclonal antibody Ec6C10 may be used to recognize cadherins expressed locally by specific endothelial cells.

Brief Summary Text (191):

A targeting vector directed toward thrombotic material in the plaque may be used to differentiate between active and inactive regions of atherosclerotic plaque. Active plaques in the process of generating thrombi are more dangerous as these plaques

may ultimately occlude a vessel or result in emboli. In this regard, in addition to low molecular weight heparin fragments, other targeting vectors, such as, for example, antifibrin antibody, tissue plasminogen activator (t-PA), anti-thrombin antibody, and fibrin antibodies directed to platelet activation factions, may be used to target active plaque with evolving clots. Most preferred targeting vectors are those which will target a plasma membrane associated GPIIb/IIIa in activated platelets in addition to targeting P-selectin, and an antibody or associated antibody fragment directed to GPIIb/IIIa. The present invention is also useful for detecting regions of acute myocardial infarction. Conveniently, by attaching antimyosin (particularly cardiomyosin) antibody or anti-actin antibodies to the polymers, infarcted myocardium may be detected by the methods of the present invention. For targeting to granulation tissue (healing wounds), many of the above targeting vectors may be useful. The wound healing tripeptide, arginine-glycine-aspartic acid (RGD), may also be used as a targeting vector in this regard.

Brief Summary Text (195):

There are a variety of cell surface epitopes on epithelial cells for which targeting vectors may be selected. For example, the protein human papilloma virus (HPV) has been associated with benign and malignant epithelial proliferations in skin and mucosa. Two HPV oncogenic proteins, E6 and E7, may be targeted as these may be expressed in certain epithelial derived cancers, such as cervical carcinoma. See Curr. Opin. Immunol. Vol. 6 (5), pp. 746-54 (1994). Membrane receptors for peptide growth factors (PGF-R), which are involved in cancer cell proliferation, may also be selected as tumor antigens. See Anticancer Drugs, Vol. 5 (4), pp. 379-93 (1994). Also, epidermal growth factor (EGF) and interleukin-2 may be targeted with suitable targeting vectors, including peptides, which bind these receptors. Certain melanoma associated antigens (MAA), such as epidermal growth factor receptor (EGFR) and adhesion molecules (Tumor Biol., Vol. 15 (4), pp. 188-202 (1994)), which are expressed by malignant melanoma cells, can be targeted with the polymer-chromophore compounds and compositions provided herein. The tumor associated antigen FAB-72 on the surface of carcinoma cells may also be selected as a target.

Brief Summary Text (196):

A wide variety of targeting vectors may be selected for targeting myocardial cells. Exemplary targeting vectors include, for example, anticardiomyosin antibody, which may comprise polyclonal antibody, Fab'2 fragments, or be of human origin, animal origin, for example, mouse origin, or of chimeric origin. Additional targeting vectors include dipyridamole; digitalis; nifedipine; apolipoprotein; low density lipoproteins (LDL), including alpha-LDL, vLDL and methyl LDL; ryanodine; endothelin; complement receptor type 1; IgG Fc; beta 1-adrenergic; dihydropyidine; adenosine; mineralocorticoid; nicotinic acetylcholine and muscarinic acetylcholine; antibodies to the human alpha 1A-adrenergic receptor; bioactive agents, such as drugs, including the alpha 1-antagonist prazosin; antibodies to the anti-betareceptor; drugs which bind to the anti-beta-receptor, anticardiac RyR antibodies; endothelin-1, which is an endothelial cell-derived vasoconstrictor peptide that exerts a potent positive inotropic effect on cardiac tissue (endothelin-1 binds to cardiac sarcolemmal vesicles); monoclonal antibodies which may be generated to the T cell receptor alpha-beta receptor and thereby employed to generate targeting vectors; the complement inhibitor sCR1; drugs, peptides or antibodies which are generated to the dihydropyridine receptor; monoclonal antibodies directed towards the antiinterleukin 2 receptor may be used as targeting vectors to direct the present chromophore polymer compounds and compositions to areas of myocardial tissue which express this receptor and which may be up-regulated in conditions of inflammation; cyclosporine for similarly directing the compositions to areas of inflamed myocardial tissue; methylisobutyl isonitrile; lectins which bind to specific sugars on membranes of cardiac myocytes and cardiac endothelial cells; adrenomedullin (ADM), which is an endogenous hypotensive and vasorelaxing peptide; atrial natriuretic peptide (ANP); C-type natriuretic peptide (CNP), which is a 22 amino acid peptide of endothelial cell origin and is structurally related to atrial natriuretic peptide but genetically distinct, and possesses vasoactive and antimitogenic activity; vasonatrin peptide (VNP) which is a chimera of atrial natriuretic peptide (ANP) and C-type nathuretic peptide (CNP) and comprises 27 amino acids; thrombin; endothelium derived relaxing factor (EDRF); neutral endopeptidase 1 (NEP-1); competitive inhibitors to EDRF, including, for example, NG-monomethyl-L-arginine (L-NNMA); potassium channel antagonists, such as charybdotoxin and glibenclanmide; antiheart antibodies, which can be identified in patients with idiopathic dilated cardiomyopathy but which preferably do not elicit cytolysis in the myocardium; antibodies directed against the adenine nucleotide translocator, the branched-chain keto acid dehydrogenase or cardiac myosin; specific antagonists for the endothelin-A receptor, which may be referred to as BQ-123; and antibodies to the angiotensin II receptor.

Brief Summary Text (197):

Two of the major antigens of heart muscle fiber sarcolemma are calcium binding glycoproteins which copurify with the dihydropyridine receptor. Antisera may be raised, including polyclonal or monoclonal antibodies, against purified sarcolemma. These antibodies may also be employed as targeted vectors. Purified fractions of the calcium binding glycoproteins may be isolated from the plasma membranes of the sarcolemma and then used to generate antibodies. ANP, which, as noted above, may be used as a targeting vector, can be obtained from cultures of human aortic endothelial cells. ANP is generally localized in endothelium, but also may localize to the endothelial or myocardial tissue. ANP may be prepared, for example, using recombinant techniques, as well as by synthesis of the peptide using peptide synthesis techniques well known to those skilled in the art. It is also possible to use an antibody, either polyclonal or monoclonal, directed towards ANP. Similarly, a peptide directed to ANP may be used for targeting endothelial and/or myocardial cells. Both the beta and alpha forms of atrial natriuretic factor may be used as potential targeting vectors for directing the present polymer chromophore compounds and compositions to myocardial tissue.

Brief Summary Text (247):

In accordance with preferred embodiments, the targeting vectors may be linked or attached to the chromophores and linking groups of the polymers of this invention via a linking group. A variety of linking groups are available and would be apparent to one skilled in the art once armed with the present disclosure. Preferably, the linking group comprises a hydrophilic polymer. Suitable hydrophilic linker polymers include, for example, polyvinylpyrrolidones, poly(vinyl methyl ethers), polyacrylamides, such as, for example, poly(methacrylamides), poly(N,Ndimethylacrylamides) and poly(hydroxypropylmethylamides), poly(hydroxyethyl acrylates), polyhydroxypropyl methacrylates, polymethyloxazolines, polyethyloxazolines, polyhydroxyethyloxazolines, polyhydroxypropyloxazolines, polyvinyl alcohols, polyphosphazenes, poly(hydroxyalkylcarboxylic acids), polyoxazolidines, and polyaspartamide. The hydrophilic polymers are preferably selected from the group consisting of polyvinylalcohol and polyvinylpyrrolidone and copolymers thereof. The hydrophilic polymer used as a linking group is preferably a bifunctional polymer. In this case, one end of the hydrophilic polymer is linked to a chromophore or linking group of the polymer of this invention, and the other end of the hydrophilic polymer is linked to the targeting vector, for example, via an amide bond. A hydrophilic polymer substituted with a terminal carboxylic acid group on one end and an terminal amino group on the other end may also be used. These latter bifunctional hydrophilic polymers may be preferred in some embodiments since they possess various similarities to amino acids. Standard peptide methodology may be used to link the targeting vector to the chromophore or linking groups of the polymer of this invention. Bifunctional hydrophilic polymers may be synthesized using standard organic chemistry methodologies. In addition, many of these materials are available commercially. An advantage of using a hydrophilic polymer material as a linking group is that the size of the polymer can be varied such that the number of monomeric subunits may be as few as, for example, about 5, or as many as, for example, about 500 or even greater. Accordingly, the "tether" or length of

the linkage may be varied as desired. This may be important, depending, for example, on the particular targeting vector employed. For example, a targeting vector which comprises a large protein molecule may require a short tether, such that it will simulate a membrane bound protein. A short tether would also allow the chromophore polymer to reside proximal to the cell of interest and thus facilitate further chromophore-polymer to cell interaction such as hydrophobic chromophore in the polymer to lipid or protein in the cell membrane.

Brief Summary Text (250):

Further examples of linking groups are polymeric units. As used herein, a polymeric unit may be of natural, semi-synthetic (modified natural) or synthetic origin. The term "polymeric unit" denotes a compound comprised of two or more repeating monomeric units, and preferably 10 or more repeating monomeric units. The phrase semi-synthetic polymeric unit (or modified natural polymeric unit), as employed herein, denotes a natural polymer unit that has been chemically modified in some fashion. Exemplary natural polymeric units suitable for use in the present invention include naturally occurring polysaccharides. Such polysaccharides include, for example, arabinans, fructans, fucans, galactans, galacturonans, glucans, mannans, xylans (such as, for example, inulin), levan, fucoidan, carrageenan, galatocarolose, pectic acid, pectins, including amylose, pullulan, glycogen, amylopectin, cellulose, dextran, dextrin, dextrose, polydextrose, pustulan, chitin, agarose, keratan, chondroitan, dermatan, hyaluronic acid, alginic acid, xanthan gum, starch and various other naturalhomopolymer or heteropolymers, such as those containing one or more of the following: aldoses, ketoses, acids or amines: erythrose, threose, ribose, arabinose, xylose, lyxose, allose, altrose, glucose, mannose, gulose, idose, galactose, talose, erythrulose, ribulose, xylulose, psicose, fructose, sorbose, tagatose, mannitol, sorbitol, lactose, sucrose, trehalose, maltose, cellobiose, glycine, serene, threonine, cysteine, tyrosine, asparagine, glutamine, aspartic acid, glutamic acid, lysine, arginine, histidine, glucuronic acid, gluconic acid, glucaric acid, galacturonic acid, mannuronic acid, glucosamine, galactosamine, and neuraminic acid, and naturally occurring derivatives thereof. Accordingly, suitable polymers include, for example, proteins, such as albumin. Exemplary semi-synthetic polymeric units include carboxymethylcellulose, hydroxymethylcellulose, hydroxypropylmethylcellulose, methylcellulose, and methoxycellulose. Exemplary synthetic polymeric units suitable for use in the present invention include polyethyleneglycol (PEG), polymethylenes (such as, for example, polyethylene and also polyethylene terephthlate), polypropylenes (such as, for example, poly(propylene glycol)), polyurethanes, poly (vinyl acetate), partially hydrolyzed polyvinyl acetate (such as, for example, poly (vinyl acetate-co-vinyl alcohol)), poly(vinyl alcohol) such as, for example, polyvinyl alcohol (PVA), poly(vinyl chloride), poly(N-vinylpyrrolidone), polyamides including nylon polyamides, polystyrene, poly(3-methoxystyrene), poly(4methoxystyrene), poly(3,4-dimethoxystyrene), poly(3,4-methylenedioxystyrene), polylactic acids, fluorinated hydrocarbons, fluorinated carbons (such as, for example, polytetrafluoroethylene), and polymethylmethacrylate, and derivatives thereof. Preferred are biocompatible synthetic polymeric units or copolymeric units prepared from monomers, such as ethylene, acrylic acid, methacrylic acid, ethyleneimine, crotonic acid, acrylamide, ethyl acrylate, methyl methacrylate, 2hydroxyethyl methacrylate (HEMA), lactic acid, glycolic acid, E-caprolactone, acrolein, cyanoacrylate, bisphenol A, epichlorohydrin, hydroxyalkylacrylates, siloxane, dimethylsiloxane, ethylene oxide, propyleneoxide, ethylene glycol, hydroxyalkylmethacrylates, N-substituted acrylamides, N-substituted methacrylamides, N-vinyl-2-pyrrolidone, 2,4-pentadiene-1-ol, vinyl acetate, acrylonitrile, styrene, p-aminostyrene, p-aminobenzylstyrene, p-vinylbenzylamine, sodium styrene sulfonate, sodium 2-sulfoxyethylmethacrylate, vinylpyridine, 2methyl-5-vinyl pyridine, 2-vinyl pyridine, 4-vinyl pyridine, aminoethyl methacrylates, 2-methacryloyloxytrimethylammonium chloride, and polyvinylidene, as well polyfunctional crosslinking monomers such as N,N'-methylenebisacrylamide, ethylene glycol dimethacrylates, 2,2'-(p-phenylenedioxy)diethyl dimethacrylate, divinylbenzene, triallylamine and methylenebis-(4-phenylisocyanate), including

combinations thereof. Preferable polymeric units include poly(acrylic acid), poly (ethyleneimine), poly(methacrylic acid), poly(methyl methacrylate), polysiloxane, polydimethylsiloxane, polylactic acid, poly(E-caprolactone), epoxy resin, melamines, triazinamine, triamino-s-triazine and polyamide (nylon) polymers. Preferable copolymer units include the following: polyvinylidene-polyacrylonitrile-poly(methyl methacrylate), polystyrene-polyacrylonitrile and poly d,l-lactide-coglycolide polymers.

Brief Summary Text (287):

Where the particles contain other components besides the contrast agent compound, e.g., matrix or <u>membrane</u> forming materials, coating agents, solvents, gases or gas generators, etc, these will conveniently be materials which are physiologically tolerable at the dosages used. The formation of droplets, coated particles, composite particles, vesicles, etc is well described in the literature, especially that relating to pharmaceutical and contrast agent (e.g., ultrasound contrast agent) preparation and formulation.

Brief Summary Text (288):

Where a water-soluble contrast agent is to be used to mark tumors for surgical removal, then it may be preferred to administer the agent in particle form in order to reduce staining of, and hence removal of, healthy tissue surrounding the tumor. Indeed in place of conventional water-soluble chromophores such as methylene blue, it may be desirable to use derivatised analogs, where derivatisation has been to introduce lipophilic groups which will reduce leakage of the chromophore from the particle, e.g., an emulsion droplet or a liposome. For this purpose, one may use long chain hydrophobic groups, such as C.sub.10-30 alkyl or alkenyl chains, preferably groups of a similar length to the lipophilic component of any liposome membrane forming material that is present in the particle. This type of chromophore derivatization can be made for any chromophore, in particular charged chromophores, especially thiazine chromophores such as methylene blue.

Brief Summary Text (290):

Alternatively, these contrast agent compounds may be administered via the oral route for absorption through the lining of the stomach, the intestines, and the colon, see for example, Carrier-mediated intestinal transport of drugs, Tsuji, A.; Tamai, I., Pharmaceutical Research (New York) Vol. 13, No. 7, p. 963-977, 1996; Oral protein drug delivery, Wang, Wei, J. Drug Targeting Vol. 4, No. 4, 1996, pp. 195-232; Improved passive oral drug delivery via prodrugs, Taylor, Michael D., Adv. Drug Delivery Rev. Vol. 19, No. 2, 1996, pp. 131-148; Oral colon-specific drug delivery: a review, Van den Mooter, Guy; Kinget, Renaat, Drug Delivery, Vol. 2, No. 2, 1995, pp. 81-93; Present status of controlled drug delivery system-overview, Naik, S. R.; Shanbhag, V., Indian Drugs, Vol. 30, Sepetember 1993, pp. 423-429; Novel formulation strategies for improving "oral" bioavailability of drugs with poor membrane permeation or presystemic metabolism, Aungst, B. J., Journal of Pharmaceutical Sciences (USA), Vol. 82, No. October 1993, pp. 979-987; Remington's Pharmaceutical Sciences, A. Osol, ed., Mack Publishing Co. 1975, Part 6, chp 40 and references therein. (pp 731-753), Part 8, all chps (pp 1355-1644); The Extra Pharmacopoeia, Martindale, 29th Edition, The Pharmaceutical Press, London, 1989.

Brief Summary Text (291):

Administration of drugs and other agents by this route is often preferred due to enhanced patient compliance (for repeated dosing) and ease of administration. It is well known in the art that not every agent is bioavailable via this route; that is to say, that not all molecules are 1) chemically stable in the environs of the gut, 2) transportable across alimentary membranes for absorption into the blood/lymphatics, and 3) active even if accessible due to metabolic processes within the gut or possible solubility issues, etc. However, it is also known in the art, that alteration of the molecular structure to control the relative hydrophobicity of the molecule (i.e., partition coefficient between octanol and water; log(P)) within a preferred range can increase the oral availability of the

agent.

Brief Summary Text (316):

A preferred contrast agent for intraoperative CSLM, OCT, photoacoustic, acoustooptical, diffusive wave, time-resolved imaging, endoscopic, multiphoton excitation microscopy or visual observation techniques will have the following properties: it will consist of stabilized particles in aqueous or buffered solution. The particle size may be around 300 to 1300 nm (i.e., roughly equal to the wavelength of the light source). The refractive index of the particles will differ from that of body fluids such as blood and lymph by at least 0.01. The particles may be made of a chromophore polymer compound or may contain or be coated with a chromophore polymer compound, e.g., the particles may comprise a matrix material (e.g. a physiologically tolerable synthetic or non-synthetic polymer, such as an acrylate or polysaccharide) incorporating a chromophore polymer compound, a core of a chromophore polymer compound coated with a coating agent or encapsulated by a membrane forming material, or a core of a matrix material with a chromophore polymer compound coated on or attached to the particle surface. The particles may be solid, semi-solid or liquid and may be layered structures such as vesicles (e.g., micelles, liposomes and microballoons). The chromophore polymer compound used will preferably be a fluorescent material, particularly a material having an emission maximum in the near infrared range, especially in the range 650 to 900 nm. Optionally the particles may have suitable surface modifying agents, such as poly (ethylene glycol) to slow their uptake by macrophages in the body. Examples of suitable particulate agents are described in WO 96/23524. Optionally, the particles can be cells coated with the polymers of this invention. These coated cells can be formed in the body with injected agents or externally from cells extracted from the body and then injected into the body.

Detailed Description Text (3):

PEG3400 diamine (Shearwater Polymers, Huntsville, Ala.; 0.39 g) was dissolved in pyridine (75 mL) with magnetic stirring. Approximately 50 mL of pyridine was distilled off under nitrogen from an oil bath at 120-130.degree. to dehydrate the PEG. The solution was cooled to ambient temperature and ClAlPc (SO.sub.2 Cl).sub.4 (0. 11 g) prepared from the corresponding acid, Porphyrin Products, Logan, Utah) was added. The solution was stirred for 18 hours at 20.degree. and then heated to reflux for 30 minutes, and cooled. The solvent was removed on a rotary evaporator at 40.degree., and the residue was then dissolved in water. This solution was then passed successively through a strong acid (H.sup.+ form) ion exchange resin and then a strong base (Na.sup.+ form) ion exchange resin to convert the product to the Na.sup. + salt. Low molecular weight components were removed by diafiltration through a 10,000 molecular weight membrane (Amicon, Beverly, Mass.), and the dark blue residual liquid was evaporated on a rotary evaporator at 40.degree. to yield a dark blue solid (0.09 g). Size exclusion HPLC analysis indicated that the product, Polymer 1, had an average molecular weight of 150,000. Absorption wavelength: .lambda..sub.max 676 nm.

Detailed Description Text (9):

The method similar to that described in Example 1, above, but employing PEG 5000-.alpha.,.omega.-amine (Shearwater Polymers, Huntsville, Ala.; 2.50 g), pyridine (50 mL, of which about 30 mL were distilled off), and ClAlPc(SO.sub.2 Cl).sub.4 (0.10 g) was used to prepare Polymer 2. The aqueous solution was heated to reflux under nitrogen for 30 minutes, and then the solvent was removed by distillation under vacuum. The reaction mixture was diafiltered through a 10,000 molecular weight membrane. The desired product did not pass through the membrane, but was isolated as a dark blue solid (yield: 0.08 g). Absorption wavelength: .lambda..sub.max 676 nm.

Detailed Description Text (54):

This dye was made from the Surfactant T908 amino derivative (2.50 g, 0.1 mM) by a method analogous to that of Example 29 but using a tenfold excess of zinc

phthalocyanine tetrasulfonyl chloride (4.0 g, 4.1 mM). The diafiltrate retentate (10000 molecular weight membrane) was evaporated and freeze dried to yield a dark blue solid, 3.2 g, lambda max. 635 nm (shoulder at 671 nm) in water.

Detailed Description Text (57):

This was prepared by a method analogous to that used in Example 1 but using PEG 10,000 diamine (Shearwater Polymers, 1.0 g, 0.1 mM) and an excess of chloro-aluminumphthalocyanine tetrasulfonyl chloride (0.217 g, 0.22 mM). The diafiltrate retentate (3000 molecular weight membrane) was evaporated to yield a dark blue solid, 0.82 g, lambda max. 675 nm in water.

Detailed Description Text (67):

PEG 3400 diamine (Shearwater Polymers, Huntsville, Ala.; 0.391 g, 0.115 mMoles) was dissolved in pyridine (75 mL) with magnetic stirring. Approximately 50 mL pyridine were distilled off under nitrogen from an oil bath at 120-130.degree. to dehydrate the PEG, and then the solution was cooled to ambient temperature and ClAlPc (SO.sub.2 Cl).sub.4 (prepared from the corresponding acid, Porphyrin Products, Logan, Utah) added (0.111 g, 0.115 mMoles). The solution was stirred for 18 hours at 20.degree. and then refluxed for 30 minutes, after which the solvent was removed on a rotary evaporator at 40.degree. and the residue dissolved in water. This solution was then passed successively through strong acid and strong base (Na form) ion exchange resins to convert the product to the Na salt. Low molecular weight components were removed by diafiltration through a 10,000 molecular weight membrane (Amicon, Beverly, Mass.) and the dark blue residual liquid evaporated on a rotary evaporator at 40.degree. to yield a dark blue solid (0.09 g).

Detailed Description Text (72):

This was prepared by the same method used in Example 18, but using ethylenediamine (Aldrich, 0.0058 g, 0.10 mMoles) in place of the PEG diamine. The aqueous solution of the product was diafiltered through a 500 molecular weight $\underline{\text{membrane}}$, and the dark blue residual solution ion exchanged to the sodium salt, and evaporated to yield a dark blue solid (0.10 g).

Detailed Description Text (75):

The method used was similar to that described in Example 18, but using PEG 5000 .alpha.,.omega.-bis amine (Shearwater Polymers, Huntsville, Ala.; 2.50 g, 0.50 mMoles), pyridine (50 mL, of which about 30 mL were distilled off), and ClAlPc (SO.sub.2 Cl).sub.4 (0.10 g, 0.10 mMoles). The solution was refluxed under nitrogen for 30 minutes, and then the solvent was removed. Diafiltration using a 10,000 molecular weight membrane, collecting the product that did not pass through the membrane, yielded a dark blue solid (0.08 g). It had .lambda..sub.max 676 nm (water). When a solution of this compound in phosphate buffered saline was injected into female immunodeficient mice with HT-29 tumors, 2.5% of the injected dose was localized in the tumor after one hour.

Detailed Description Text (110):

A total of 1.25 g of the .alpha.,.omega.-bis-(amino) analog of Pluronic Surfactant F-108 from above (intermediate E) was treated with 0.026 g of dimethylaminopyridine and 10 m of anhydrous pyridine. The resulting solution was treated with 0.12 g of rhodamine B sulfonyl chloride (Molecular Probes) and stirred at room temperature under nitrogen overnight. The resulting intensely purple solution was stripped on a rotary evaporator to an intensely purple solid comprising 1.42 g. A total of 1.0 g of the crude product was dissolved in 40 ml of distilled water, filtered through a 0.45 micron nylon filter, and the filtrate diafiltered against distilled water using an 50 ml stirred diafiltration cell (Amicon) containing a 3,000 nominal molecular weight cellulose acetate diafiltration membrane (Amicon YM-3). The diafiltration was continued for 35 turnovers (1,750 ml of diafiltrate removed). Initially, the diafiltrate was intensely purple, but as the purification continued the color intensity diminished till it was virtually colorless at 35 turnovers. The intensely purple retentate was stripped on a rotary evaporator to an intensely

purple solid which comprised 0.92 g (final product F). The .sup.13 C NMR spectrum of the product contains the dominant polyalkylene oxide peaks between 70 and 80 ppm seen in F-108 and all the subsequent intermediates, as well as a new singlet at 45.69 ppm. No remaining peak near 41 ppm, corresponding to the previous bis-amine intermediate, was observed. Size exclusion HPLC studies indicate a single broad peak with a peak molecular weight of approximately 15,000 based on PEG standards. The compound shows a broad spectral absorbance peaking at 584 nm. ##STR22##

Detailed Description Text (113):

A total of 0.30 g of the terminal amino derivative of Surfactant T908 was dissolved in 3.0 ml of distilled water and then treated with 2.9 ml of 0.5M aqueous sodium carbonate. The resulting clear solution was treated with 0.19 g of the sulfoindocyanine dye sold as Cy-7 dye by Amersham and stirred at room temperature for 18 hours. The resulting clear solution was diluted with 6.0 ml of distilled water and placed in a stirred diafiltration cell equipped with an Amicon YM-10 (nominal 10K cutoff) diafiltration membrane. It was diafiltered until 250 ml of diafiltrate had been removed (21 turnovers). The retentate was the freeze dried to a fluffy polymeric solid comprising 0.21 g Spectral analysis showed the polymer has a lambda max. (absorption) of 741 nm.

Detailed Description Text (121):

A total of 1.00 g of the above terminal amino derivative of Surfactant T908 was dissolved in 10 ml of distilled water and then treated with 3.2 ml of 0.5M aqueous sodium carbonate. The resulting clear solution was treated with 0.156 g of fluorescein isothiocyanate, isomer I (Aldrich) and stirred at room temperature for 18 hours. The resulting clear intensely orange solution was diluted with 60 ml of distilled water and placed in a stirred diafiltration cell fitted with an Amicon YM-10 (nominal 10K cut-off diafiltration membrane. It was initially ultrafiltered down to a retentate volume of 25 ml and was subsequently diafiltered until 600 ml of diafiltrate had been removed (24 turnovers). The retentate was then freeze dried to a bright orange fluffy polymeric solid comprising 0.43 g. Spectral analysis showed the polymer has a lambda max. (absorption) of 307 nm.

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1. Document ID: US 6897072 B1

L23: Entry 1 of 2

File: USPT

May 24, 2005

US-PAT-NO: 6897072

DOCUMENT-IDENTIFIER: US 6897072 B1

TITLE: Probes for a gas phase ion spectrometer

DATE-ISSUED: May 24, 2005

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Rich; William E. Redwood Shores CA
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Yip; Tai-Tung Cupertino CA

Beecher; Jody San Jose CA

US-CL-CURRENT: 436/173

Full Title Citation Front Review Classification Date Reference

2. Document ID: US 6881364 B2

L23: Entry 2 of 2 File: USPT Apr 19, 2005

US-PAT-NO: 6881364

DOCUMENT-IDENTIFIER: US 6881364 B2

TITLE: Hydrophilic mixed matrix materials having reversible water absorbing

properties

DATE-ISSUED: April 19, 2005

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Vane; Leland Morris Cincinnati OH Ponangi; Ravi Prasad Modesto CA

US-CL-CURRENT: <u>264/41</u>; <u>264/216</u>, <u>264/236</u>, <u>264/237</u>

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L25: Entry 6 of 9

File: USPT

Feb 25, 1992

DOCUMENT-IDENTIFIER: US 5091080 A

** See image for <u>Certificate of Correction</u> **

TITLE: Adsorbents for the removal of volatile substances from aqueous systems

Abstract Text (1):

Semipermeable membrane-enclosed solid core adsorbent devices for the selective removal of volatile chemical species from an aqueous environment are disclosed.

Brief Summary Text (3):

Ammonia has been stripped from aqueous streams by conventional packed towers, but has been criticized for releasing ammonia to the atmosphere and often results in fouling of the packing materials. Polar organic solvents were reported to be removed from aqueous solutions by hydrophobic adsorbents of ion exchange particles in a 1987 University of California, Berkeley, Dissertation Thesis by William G. Rixley entitled "Non-Wetting Adsorbents for the Recovery of Solutes from Dilute Aqueous Solutions."

Brief Summary Text (5):

The present invention comprises an adsorbent device for the selective removal of a volatile and trappable chemical species from an aqueous environment comprising at least one non-liquid core containing at least one trapping agent, the core being continuously enclosed by a semipermeable membrane.

Detailed Description Text (2):

In accordance with the present invention there is provided a novel device and associated method for the extraction of volatile species from aqueous systems. In its simplest form, the device comprises one or more solid cores, each core containing at least one trapping agent, with the core(s) being continuously enclosed by a semi-permeable membrane. By "solid" is meant non-liquid and nongaseous, including dense and porous solids and gels. "Trapping agent" is defined to mean any anionic, cationic, reducing, oxidizing or neutral chemical species capable of neutralizing, reducing, oxidizing or complexing with the volatile and trappable species to be removed from the aqueous environment. The volatile and trappable species may be acidic, basic or neutral. "Semipermeable" is defined to mean freely permeable to gases but substantially impermeable to liquid water, such as in the case of microporous hydrophobic polymers. Dense solid cores may have the trapping agent coated or chemically bonded to their exterior, may comprise matrices within which the trapping agent is entrapped or chemically bonded, or may comprise the trapping agent itself. Especially preferred dense and porous solid cores are those of polystyrene, polyacrylate, polyamide, polyester, cellulose acetate or regenerated cellulose. Materials also preferred for porous solid cores are ceramics, silica, alumina, glass and various forms of diatomaceous earth. Porous solid cores may be filled with liquid or gel trapping agents. Gels may comprise a mixture of trapping agent, water and a hydrophilic polymer gelling agent. Exemplary gelling agents include acrylamide polymers such as polyacrylamide and partially hydrolyzed polyacrylamide; acrylic acid and methacrylic acid polymers and copolymers of maleic and acrylic acids; vinyl carboxylic acid polymers and copolymers; cellulosics such as cellulose, carboxymethyl cellulose, 2,3-di-(2,3diiodopropoxy)propylcellulose; cellulose thiocyanate, and cellulose etherpolyacrylamide aqueous gels; epoxy polymers; ethylene oxide polymers; phenolformaldehyde condensation polymers; gelatin and gelatin products; natural gums such as tamarind gum, xanthan gum, xylitol gum, galactomannan gum, and polygalactomannan allyl ether gel; polyamides such as polyamide resin, poly-(amide-imide) resin, and poly(m-phenylene isophthalamide); polyesters such as vinyl ester copolymer and vinyl acetate copoymer; polyethylenimines; polyurethanes; polyvinyl alcohols; polyvinylpyrrolidone, N-vinylpyrrolidone-vinyl alkylcarboxylate copolymer, and N-vinylpyrrolidone-alkyl acrylate copolymer; and hydrolyzed polyacrylonitrile-grafted starch. In all cases, the trapping agent may be acidic, basic, neutral, oxidizing or reducing so long as, in those cases where there is more than one type, the agents are such that they do not neutralize each other.

<u>Detailed Description Text</u> (3):

The solid trapping agent-containing cores are surrounded or completely and continuously enclosed by a semipermeable membrane in one of four different forms: (1) a coating directly on the core; (2) a capsule; (3) sealed hollow fibers; and (4) a pouch. The <u>adsorbent</u> devices may be used for the selective removal of volatile species from aqueous systems by simply contacting the aqueous system containing the undesirable species with the <u>adsorbent</u> devices. Such contact may be effected in conventional ways such as dispersing the devices in the aqueous system, in packed towers and columns, and in fluidized beds.

<u>Detailed Description Text</u> (28):

P: glucose/glucose oxidase/catalase coated on Celite.RTM. <u>particles</u> (Manville Corp., Denver, Colo.)

<u>Detailed Description Text</u> (33):

One particularly preferred embodiment of the present invention comprises highly selective ammonia-absorbent beads comprising strongly acidic cation-exchange resin covered with a supported-gas membrane. The trapping mechanism and methods of fabrication for such membrane-coated beads is essentially the same for all five types of beads and particles disclosed above. The membrane consists of a gas layer about 100 microns thick that separates the aqueous ammonia-containing solution from the strongly acidic cation-exchange beads. The gas layer is supported and stabilized within the pores of a hydrophobic or non-water-wettable polymer film that is coated on the cation-exchange bead. Since water cannot wet the polymer film, the ammonia solution cannot contact the strongly acidic cation-exchange bead. Permeation through the supported-gas membranes is very selective in that only volatile solutes such as ammonia pass through the membrane, while nonvolatile solutes are completely rejected.

<u>Detailed Description Text</u> (36):

The microporous membrane-coated beads of the present invention may be fabricated by coating bead or particle cores with a hydrophobic polymer or polymer blend solution consisting of polymer, solvent, and a nonsolvent. Especially preferred hydrophobic polymers include polyethylene (PE), polypropylene (PP), polysulfone (PS), polyethersulfone (PES), poly(vinylidene difluoride) (PVDF), poly (tetrafluoroethylene) (PTFE), and compatible mixtures thereof. Other suitable polymers useful for the hydrophobic bead or core coating are set forth in Table 2, including blends thereof and blends thereof with perfluoro compounds. The membrane is formed by coating the bead with a wet coat of polymer in solvent followed by immersing the coated bead in a quench bath from -70.degree. C. to +100.degree. C. where the solvent exchanges with the quench solution, causing the polymer to undergo phase separation and precipitate (phase inversion) around the bead. The porosity, thickness, and pore-size distribution of the resulting membrane can be controlled by adjustments of process parameters such as quench bath composition, temperature of the polymer solution and/or the quench bath, and the composition of the polymer solution. Generally speaking, porosity and pore size increase with an increase in solvent concentration in the quench bath, with an increase in nonsolvent concentration in the polymer solution and an increase in temperature in the polymer solution, with an increase in the temperature of the quench bath, with

a decrease in polymer concentration in the polymer solution, with the addition of "pore-formers" such as glycerol to the polymer solution, and with the inclusion of two or more polymers in the polymer solution that phase separate upon precipitation. Thickness of the membrane can be increased by increasing the viscosity of the polymer solution, by increasing the spray rate of the polymer solution, and by decreasing the spray rate of the atomizing air.

Detailed Description Text (38):

Cores consisting of gels and a trapping agent may be made by swelling commercially available gels with a solution containing a trapping agent. For example, polyacrylamide beads (e.g., Bio-Gel P-6, Bio-Rad Laboratories, Richmond, Calif.) can be purchased in the dry state and then swollen in an aqueous solution containing a trapping agent to form a gel. The trapping agent may be an acid (e.g., H.sub.2 SO.sub.4), a base (e.g., NaOH), or an organic compound (e.g., ethanolamines). Once swollen, the polyacrylamide gel consists of greater than 80 wt % of the aqueous solution and still remains as discrete non-agglomerating beads. These swollen beads may then be coated with a semipermeable membrane coating in the same manner as described below in Example 3. Such cores may also be made from a solution containing water, a trapping agent, and the gelling material. For example, gelling materials such as gelatin or polyvinyl alcohol may be dissolved in aqueous solutions containing a trapping agent and then dripped or sprayed into a drying chamber to form discrete gel particles or beads as water is evaporated from the solution and/or as the temperature of the solution is lowered. Trapping agents may be water-soluble or suspended in a solution containing water and a gelling material. These particles may then be coated with a semipermeable coating as described in Example 3. In addition, the solution containing water, a trapping agent, and the gelling material may be dripped or sprayed directly into the polymer coating solution as described in Example 3. Once coated with the polymer solution, the coated cores may be dried, forming a gel in the core as the solution cools and water evaporates.

Detailed Description Text (44):

Dowex MSC-1 resin was incorporated into a polyethylene (PE) matrix to minimize the incompatibility between the resin and the PS coating. The resin was crushed to a fine powder and mixed with a solution of 15 wt % PE (Tenite 808, Eastman Chemicals, Kingsport, Tenn.) dissolved in olive oil at 130.degree. C. This mixture was sprayed out of an air-atomizing nozzle into a water bath at 20.degree. C. which caused the polymer and solvent to phase separate and the polymer to precipitate. The olive oil was washed out of the particles by immersing the particles in acetone, leaving a microporous matrix of PE with resin entrapped therein. The particles were then air-dried for 16 hours. The use of the microporous PE particles allowed the resin in the particles to be mixed in a slurry with the PS coating solution. The slurry was pumped to an air-atomizing nozzle which broke the slurry up into droplets that were sprayed into a water quench bath at 20.degree. C. The PS precipitated in the water quench bath, forming a coating around the resin-containing microporous PE particles.

Detailed Description Text (46):

Water-soaked Dowex MSC-1 resin was dripped into a polymer coating solution comprising 20 wt % PS dissolved in DMAC. Due to the water impregnated in the resin, the polymer precipitated at the surface of resin particles. Since the particles were in an environment of excess polymer, i.e., the polymer solution, any defects in the coating were quickly covered by the readily available polymer. The beads were sieved from the polymer solution before the coating could redissolve with a wire screen and an air stream was used to blow excess resin off the screen. The coated particles were then placed in water at 20.degree. C. for 14 hours to wash the remaining solvent from the coatings. The so-prepared beads were tested for leaks in the membrane coating by suspending them in salt solution and monitoring increases in the hydrogen ion concentration, which would result from the exchange of H.sup.+ from the beads by Na.sup.+ leaking through the membrane. Typically, 0.5

g of coated beads (76 wt % cation-exchange resin) was suspended in 10 ml of 0.5M aqueous sodium chloride and pH was monitored as a function of time. During the first 24 hours the pH dropped from approximately 7 to 4.4 (4.times.10.sup.-4 mmol of H.sup.+ exchanged due to leakage); the pH remained constant for a week thereafter. In a control experiment using 0.5 g of uncoated cation-exchange beads, the pH dropped instantaneously from approximately 7 to 1.1 (0.79 mmol of H.sup.+ exchanged, amounting to the total ion exchange capacity for this amount of uncoated beads), indicating that the membrane-coated beads were 99.9% free of leaks.

<u>Detailed Description Paragraph Table</u> (1):	
TABLE 1	Volatile Trapping Agents Species
Class Types Applications	Ammonia acidic A,
B, mammalian cell C, F culture G, H aquacultur	e fermentation diapers (odor)
removal) blood detoxifi- cation waste stream t	reatment Hydrogen cyanide basic D, E,
food processing I, J, (e.g., almonds and L pea	ches) reactions catalyzed by the
enzyme oxynitrilase waste treatment Carbon Dio	xide basic D, E, aquaculture I, J,
analytical methods L, S to obtain total organi	c carbon carbon beverages (e.g., beer
and wine Hydrogen sulfide basic D, E, water tr	eatment I, J, removal of L, S
undesirable odors Alkyl sulfides basic D, E, a	nd tastes from I, J, foods and
beverages L, S removal of sulfur Sulfur dioxid	e basic D, E, dioxide preserva- I, J,
tive in foods and L, S beverages Carboxylic ac	ids basic D, E, deacidifying foods I,
J, and beverages K, L removal of undesirable o	dors and tastes from foods and
beverages waste treatment fermentation product	recovery Aldehydes and neutral I, J,
fermentation ketones L, M, foods and beverages	N nicotinamide cofactor regeneration
reduction reactions catalyzed by alcohol dehyd	rogenases Oxygen neutral O, P, food
processing Q, R beverages (e.g., beer and wine	
anaerobic fermentations aqueous high performan	ce liquid chromatography solvents
Halogens neutral D, E, waste treatment M dechl	
neutral T nicotinamide cofactor regeneration o	xidation reactions catalyzed by
alcohol dehydrogenase	·

CLAIMS:

- 7. An absorbent device for the selective removal of a volatile and trappable chemical species from an aqueous environment comprising at least one core containing at least one trapping agent capable of trapping said chemical species by irreversible reaction with the same, said core being enclosed by a semipermeable supported-gas membrane pouch and being selected from a dense or porous solid of polystyrene, polyacrylate, polyamide, polyester, cellulose acetate and regenerated cellulose.
- 17. The device of claim 1 wherein said trapping agent is selected from glucose, glucose oxidase, glucose catalase, ferrous sulfate, and a metallo Schiff base complex and is coated on particles of diatomaceous earth.

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<u>L25</u>	L19 and particles	9	<u>L25</u>
<u>L24</u>	L15 and particles	. 0	<u>L24</u>
<u>L23</u>	L20 and silicon oxide	2	<u>L23</u>
<u>L22</u>	L21120 and silicon dioxide	0	<u>L22</u>
<u>L21</u>	L20 and carbon	15	<u>L21</u>
<u>L20</u>	L19 and adsorbent	18	<u>L20</u>
<u>L19</u>	L18 and polyvinyl alcohol	18	<u>L19</u>
<u>L18</u>	L13 and aldehyde	45	<u>L18</u>
<u>L17</u>	adsorvent an amine and aldehyde and polyvinylalcohol	0	<u>L17</u>
<u>L16</u>	polyalkylamine and adehydes and poglycol and croslink?	0	<u>L16</u>
<u>L15</u>	adsorvent and polyalkylamine and pva and particle and crosslinker	. 0	<u>L15</u>
<u>L14</u>	membrane and amine same pva and aldehyde and adsorbent	2	<u>L14</u>
<u>L13</u>	adsorbent and polyamide same membrane	264	<u>L13</u>
<u>L12</u>	L9 and polyallyl	4	<u>L12</u>

<u>L11</u>	L9 and polyallyl same amine	0	<u>L11</u>
<u>L10</u>	L9 and polyalkylamine	0	<u>L10</u>
<u>L9</u>	L8 and aldehyde	104	<u>L9</u>
<u>L8</u>	ll and crosslink	259	<u>L8</u>
<u>L7</u>	11 and croslinker	0	<u>L7</u>
<u>L6</u>	11 and interfaceial polymerization	0	<u>L6</u>
<u>L5</u> .	L3 and interfacial	0	<u>L5</u>
<u>L4</u>	L3 and crosslink?	0	<u>L4</u>
<u>L3</u>	L2 and 210/502.1.ccls.	7	<u>L3</u>
<u>L2</u>	L1 and sorbent	54	<u>L2</u>
<u>L1</u>	membrane and polyamide and filler	3090	L1

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<u>L33</u>	L28 and crosslinking	1	<u>L33</u>
<u>L32</u>	L28 and cross-linking	0	<u>L32</u>
<u>L31</u>	L28 and cross-linker	0	<u>L31</u>
<u>L30</u>	L28 and crosslinker	0	<u>L30</u>
<u>L29</u>	L28 and aldehyde	0	<u>L29</u>
<u>L28</u>	L27 and silicon dioxide	1	<u>L28</u>
<u>L27</u>	polyamine same polyvinylalcohol and activate carbon and particles	1	<u>L27</u>
<u>L26</u>	pyamine same polyvinylalcohol and activate carbon and particles	0	<u>L26</u>
<u>L25</u>	L19 and particles	9	<u>L25</u>
<u>L24</u>	L15 and particles	0	<u>L24</u>
<u>L23</u>	L20 and silicon oxide	2	<u>L23</u>
<u>L22</u>	L21120 and silicon dioxide	0	<u>L22</u>
<u>L21</u>	L20 and carbon	15	<u>L21</u>
<u>L20</u>	L19 and adsorbent	18	<u>L20</u>

<u>L19</u>	L18 and polyvinyl alcohol	18	<u>L19</u>
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<u>L17</u>	adsorvent an amine and aldehyde and polyvinylalcohol	0	<u>L17</u>
<u>L16</u>	polyalkylamine and adehydes and poglycol and croslink?	0	<u>L16</u>
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<u>L6</u>	11 and interfaceial polymerization	0	<u>L6</u>
<u>L5</u>	L3 and interfacial	0	<u>L5</u>
<u>L4</u>	L3 and crosslink?	0	<u>L4</u>
<u>L3</u>	L2 and 210/502.1.ccls.	7	<u>L3</u>
<u>L2</u>	L1 and sorbent	54	<u>L2</u>
L1	membrane and polyamide and filler	3090	L1

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L13: Entry 8 of 264

File: USPT

Apr 19, 2005

DOCUMENT-IDENTIFIER: US 6881364 B2

TITLE: Hydrophilic mixed matrix materials having reversible water absorbing

properties

Detailed Description Text (12):

The mixed matrix materials described in the present invention were fabricated using commercially available chemicals namely: PVA, 99% hydrolyzed; Poly (allylamine hydrochloride); glutaric dialdehyde (glutaraldehyde), 50% by wt. solution in water, and maleic acid 99%. Two types of backing materials were used for composite membrane preparation—1) METRICEL POLYPRO, a porous, mixed cellulosic, ester material sold by Gelman Sciences, using a 0.1 micron pore size and polyamide AK membranes obtained from Osmonics Corporation, USA. The polyamide membranes in this case being asymmetrical in nature. The colloidal silica product was obtained from Nissan Chemical Industries, Ltd. (USA). under the names: SNOWTEX-O, 40, and UP. SNOWTEX-O is a clear, aqueous, colloidal silica sol having a pH of 2-4 and containing 21.5% by wt. nano sized particles (10-20 nanometers) of silicon dioxide dispersed in water.

<u>Detailed Description Text</u> (21):

The mixed matrix membranes described in the present disclosure comprise certain organic polymer materials having a solid particulate <u>adsorbent</u> incorporated therein. In the preferred embodiment of the invention, the organic polymer material will be selected from the group of materials having affinity for water. The solid particulate <u>adsorbent</u> material which is incorporated in the hydrophilic organic polymers will be nano sized materials, which also possess hydrophilic characteristics.

Detailed Description Text (22):

The mixed matrix membrane which is prepared according to the process of the present invention possesses the ability to effect the separation of various components of a fluid (particularly water) from alcohols present in a feed mixture by utilizing the differences in the steady state permeability characteristics of each component of the mixture. The desired separation effect is enhanced by incorporating an adsorbent with certain polymeric materials.

Detailed Description Text (41):

The separating layer of the composite <u>membrane</u> contains a mixture of poly (vinyl alcohol) and poly allylamine hydrochloride) with nano sized silicon dioxide particles dispersed throughout the <u>membrane</u> matrix and crosslinked using glutaraldehyde. The backing used for preparing the composite <u>membranes</u> is commercially available <u>polyamide</u> reverse osmosis <u>membrane</u> obtained from Osmonics. The dry composite film was cross linked at 150.degree. C. for 12 minutes resulting in a separating layer having the composition as shown in table 4.

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